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In re the Application of:

J. UKON et al.

Application No.: 09/986,656

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Atty. Docket No.: 103120-00027

For: METHOD FOR MONITORING AND/OR CONTROLLING THE STATUS OF A PLASMA IN A PLASMA SPECTROMETER AND SPECTROMETER FOR IMPLEMENTING SUCH A METHOD

CLAIM FOR PRIORITY

Commissioner for Patents
Washington, D.C. 20231

February 25, 2002

Sir:

The benefit of the filing dates of the following prior foreign applications in the following foreign country is hereby requested for the above-identified patent application and the priority provided in 35 U.S.C. §119 is hereby claimed:

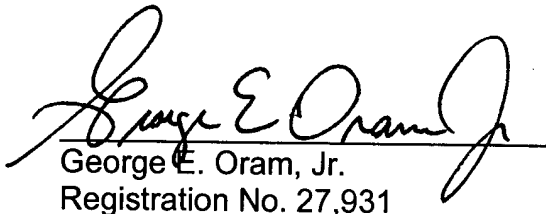
European Patent Application No. 00 403139.9 filed on November 10, 2000

In support of this claim, a certified copy of said original foreign application is filed herewith.

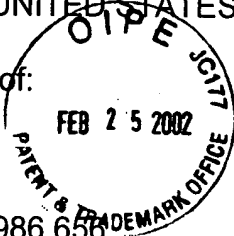
It is requested that the file of this application be marked to indicate that the requirements of 35 U.S.C. §119 have been fulfilled and that the Patent and Trademark Office kindly acknowledge receipt of these document.

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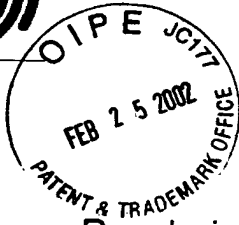




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Bescheinigung

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Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

00403139.9

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

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Sheet 2 of the certificate
Page 2 de l'attestation

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Demande n°:

Anmeldetag:
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Anmelder:
Applicant(s):
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Bezeichnung der Erfindung:
Title of the invention:
Titre de l'invention:

Method for monitoring and/or controlling the status of a plasma in a plasma spectrometer and spectrometer for implementing such a method

In Anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s) revendiquée(s)

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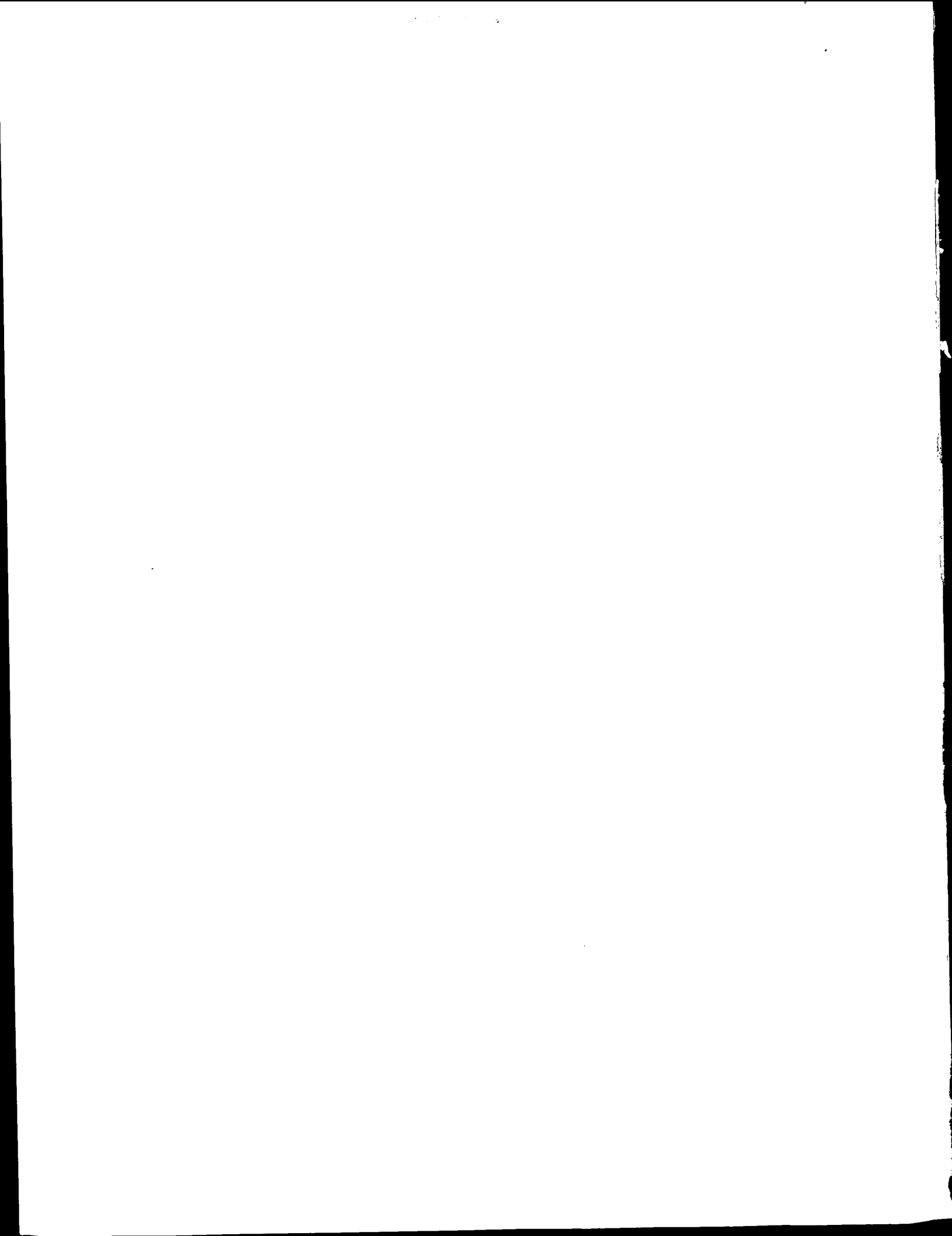
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METHOD FOR MONITORING AND / OR CONTROLLING THE
STATUS OF A PLASMA IN A PLASMA SPECTROMETER AND
SPECTROMETER FOR IMPLEMENTING SUCH A METHOD

5

BACKGROUND OF THE INVENTION

The present invention relates to a method for monitoring and/or controlling the status of a plasma in a plasma-spectrometer such as a plasma-optical emission spectrometer or atomic spectrometer (ICP-OES or ICP-MS), and to spectrometer for implementing such a method.

10

It is a very common practice to analyse a material sample, using so-called "plasma-optical emission spectrometer" such as in particular inductively coupled high frequency plasma-optical emission spectrometers (ICP-OES) or atomic spectrometers such as a mass-spectrometer, in particular inductively coupled high frequency plasma mass spectrometer

15

(ICP-MS).
In all these devices, there is obtained a plasma of the material sample to be analysed and either the light generated by the plasma or the chemical species formed in the plasma are analysed through an optical emission spectrometer or a mass spectrometer.

20

The accuracy of the analysis is highly dependent upon the plasma status and its generation conditions.

By the plasma status there is meant plasma physical characteristics such as the plasma position, in particular relative to a spectrometer entrance slit, plasma shape and/or contour, plasma intensity and/or color and in particular the position of the most intense portion of the plasma as well as the fluctuations of those characteristics.

25

In particular, the plasma position and the plasma shape and/or contour are or great influence onto the analytical performances of the spectrometer.

30

As long as those parameters are stable, the same performance can be obtained. However, those parameters are influenced by the plasma torch structure and the gas feeding rate. It means that, when the spectrometer is different, even of the same type and even with the same feeding conditions, the plasma characteristics may largely vary.

Presently, operators have to look at the plasma in the torch box, standing in front of the instrument when the conditions have changed, and usually visually determine whether the plasma is in optimal condition or not.

5 Also, the stability of the plasma influences the accuracy of the analysis data. Some spectrometers are equipped with means for showing the position of the torch mount which are associated with a scale or used with the motorised plasma torch holder. However, the scale or the position of the motorized holder does not always indicate the position of the plasma.
10 Moreover, the fluctuation of the plasma torch cannot be monitored from the holder position. Thus, even though analysis data fluctuation can be estimated as being caused by plasma fluctuation, there is no evidence to show it.

15 As already indicated, the plasma position with regard to the spectrometer entrance slit deeply influences the performance of the analysis. Since up to now, there is no existing means for determining the spectrometer entrance slit position with regard to the plasma position, there is no possibility in the review of the analysis results to consider the position of the observation.

20 The shape of the plasma is affected by the gas feed condition and the torch structure.

25 The plasma shape also influences the analytical performances. The feeding gas conditions are often stored as the measurement conditions for later analysis with the same conditions or for later data review. However, when the plasma torch is replaced, it may happen that due to small changes of the torch structure the gas conditions are changed for optimisation. Since no record of the plasma image at the optimised conditions exist, there is no way for reproducing the former plasma conditions.

30 Finally, when the operators detect some troubles with the plasma such as fluctuation of the position or of the shape or generation of strange sounds, it is almost impossible to explain the situation to the manufacturer in the absence of any plasma image.

Thus, the aim of the present invention is to provide a method for monitoring and/or controlling the status of a plasma in a plasma-optical

emission spectrometer or mass spectrometer which overcomes the drawbacks of the prior art.

The invention also concerns a plasma-spectrometer which incorporates means to implement the plasma status monitoring and/or
5 controlling method.

The above goals are achieved according to the invention by providing a method for monitoring and/or controlling the status of a plasma in a plasma-spectrometer, which comprises :

- acquiring image data of the plasma ; and
- 10 - a) displaying an image of the plasma from the acquired image data on a display device, and/or
- b) storing the image data in a computer unit.

The spectrometer may be an optical emission spectrometer (OES) or a mass spectrometer (MS) and in particular an inductively coupled high
15 frequency plasma optical emission spectrometer or mass spectrometer.

In a preferred embodiment of the process of the invention, the plasma sound is also recorded and monitored through the monitoring device and/or stored as sound data in the computer central unit.

With the present invention, the status of the plasma, such as plasma
20 position, plasma shape and contour, plasma intensity and/or color can be monitored and displayed on line on the same monitoring device such as a monitor screen of the computer unit. Alternatively or simultaneously, the plasma image data can be stored and processed in the memory of the computer unit along with measured data. Processing of image data may
25 comprise obtaining intensity images, intensity contour, color contour, intensity outline and time base fluctuation of image data. Then, the image data and the measured data of the plasma can be easily reviewed later on. In addition, the plasma image data and plasma measured data can be sent to remote places so that the plasma can be monitored from a remote site.

30 This would be also the case for the recording of the plasma sound. The image data can also be processed through the computer unit for establishing an optimal data set. For example, the image data can be processed to get the most intense position within the plasma using the outline shape, and the intensity, the colour analysis and the light intensity
35 analysis such as the contour of the plasma.

The method of the present invention allows an online continuous monitoring and/or storing of the analysis results. Therefore, the fluctuation of the plasma image characteristics based on the image analysis such as the most intense position fluctuation or the plasma shape fluctuation can be calculated and displayed and/or stored. Similarly, the plasma sound, once
5 recorded, can be, if necessary processed for example to determine frequency distribution or the time domain frequency distribution changes.

All this information can be used by the operator either immediately to determine whether or not the plasma is in requisite conditions for normal
10 analysis or later on to set up optimal plasma conditions.

When the spectrometer is an optical emission spectrometer, as it is well known, the plasma may be placed in two different orientations with regard to the spectrometer optical axis. These orientations are usually referred as radial plasma and axial plasma. Radial plasma means that the
15 plasma is vertically oriented with regard to the spectrometer optical axis and is observed in a lateral direction by the spectrometer detector device. In other words, the optical axis of the spectrometer and in fact of the spectrometer detector device is perpendicular to the plasma orientation.

On the other hand, axial plasma means that the plasma is horizontally
20 oriented and is observed by the spectrometer detector device in the axial direction. In other words, the plasma is axially oriented along the optical axis of the spectrometer detector device.

In the case of a radial plasma in which the plasma is vertically oriented with regard to the optical axis of the spectrometer, imaging of the
25 plasma is effected in register with the optical axis, for example by placing a video-camera in alignment with the spectrometer optical axis. The plasma is then positioned at the focal point of the video-camera. In that case, the entrance slit of the detector device of the spectrometer is also viewed by the video-camera and can be imaged by the video-camera simultaneously with
30 the plasma. In that later case, the entrance slit of the spectrometer detector device is focused at the plasma and is also placed at a focal point of the video-camera.

Preferably, for a better imaging, the entrance slit can be illuminated. The illumination of the entrance slit can be made from the outside or the

inside, for example by using lamps inside or outside of the monochrometer of the spectrometer detector device.

With an axial plasma, imaging of the plasma is effected in a direction perpendicular to the optical axis of the spectrometer detector device.

5 In that case of course, an imaging of the entrance slit of the spectrometer detector device is obviously not possible.

This is in particular the case when the spectrometer is a mass-spectrometer wherein the chemical species generated in the plasma must entered the detector device.

10 As indicated previously, recording of the plasma sound can be made by a microphone disposed and fixed in the plasma enclosure. The microphone may be placed in any position within the enclosure wherein a good recording a the plasma sound can be obtained. In particular, the microphone may eventually be incorporated with the video-camera.

15 The invention also concerns a plasma spectrometer for implementing the above described method which comprises a video-camera fixed on an enclosure for the plasma, preferably an inductively coupled high frequency plasma, which is coupled to a display device and/or a computer unit for imaging the plasma and/or storing plasma image data.

20 Depending upon the fact that the plasma in the spectrometer would be a radial plasma or an axial plasma, the video camera would be axially or perpendicularly oriented with regard to the optical axis of the spectrometer. However, the camera optical axis is always perpendicular to the plasma axis. In the case of a radial plasma spectrometer and with the video-camera axially positioned on the optical axis of the spectrometer, there is then a possibility of imaging simultaneously the plasma and the entrance slit of the spectrometer.

25 Since in such a case the entrance slit and the plasma are placed in register positions, a slit image can, therefore, be observed and recorded. 30 When the plasma is turned on, the slit position in the plasma can then be seen by superimposing the recorded image of the entrance slit on the monitored plasma image, thereby ensuring the analysis position in the plasma. Obviously, the superimposed images can also be recorded as analytical data for further data review.

The plasma position and the plasma shape influence the analytical performances of the spectrometer. As long as those parameters are stable, the same performances can be obtained. However, those parameters are influenced by the plasma torch structure and the gas feeding rate.

5 When the spectrometer is a different one, even of the same type, and even with the same feeding conditions, the plasma characteristics are not always similar. With the method and spectrometer according to the invention, image data of the plasma can be displayed and/or stored. This provides a possible comparison between two different plasma images or
10 plasma image data. Therefore, after having recorded the best image or the best image data for the plasma and when the plasma torch is replaced or when other instruments are installed on the spectrometer, the new setting parameters can be obtained easily so that they reproduce the same plasma shape, contour, intensity and position according to the optimal image
15 characteristics displayed or recorded.

The same holds true for the recording of the plasma sound.

The invention will now be described in connection with the figures which schematically represent, respectively :

- 20 - figure 1 : an inductively coupled high frequency plasma-optical emission spectrometer of the radial type according to the invention ;
- figure 2 : an inductively coupled high frequency plasma-optical emission or mass spectrometer of the axial type, according to the invention.

Referring to figure 1, there is represented schematically an inductively coupled high frequency plasma-optical emission spectrometer according to
25 the invention. The spectrometer as typically comprises a plasma generating device 1 and a detector device 2 comprising an entrance slit 3 optically coupled with a detector 4. The spectrometer represented on figure 1 is of the radial plasma type, that is the plasma generated in the plasma device 1 is oriented perpendicularly to the optical axis (OA) of the spectrometer
30 detector device. As usual, the detector device is focused on the plasma 1. Typically, a relay lens 6 is placed between the plasma and the entrance slit 3 of the detector device 2. According to the invention, a video-camera 7 is fixed to the plasma device enclosure, so that the lens of the camera is aligned with the optical axis OA of the spectrometer detector device, so that
35 the plasma 1 is at a focal point of the video-camera 7. With such a

configuration, the video-camera 7 is able to image both the plasma and the entrance slit 3 of the spectrometer detector device 2.

In order to improve imaging of the entrance slit 3 of the spectrometer detector device 2, a lamp 5 may be placed either inside or outside of the detector device, in order to illuminate the entrance slit 3. Also, a microphone 8 is fixed to the enclosure of the plasma generating device 1 in a position allowing recording of the plasma sound. Both video-camera 7 and a microphone 8 are connected to a computer unit 9 and through this computer unit 9 to a display device 10 such as a monitoring screen. Possibly, the computer unit 9 may be connected to a local data network through lines 11.

The spectrometer just described allows to acquire image data and to display the image on the monitor as well as to process the image data through the computer unit 9. Image data can also be recorded and stored in the computer unit 9 where they can be retrieved and eventually processed when necessary along with a spectral or analytical data. These image data can also be transferred to other computers through the local network.

The just disclosed spectrometer allows to acquire image data both from the plasma and from the entrance slit 3 of the detector device 2 of the spectrometer.

There is represented on figure 2 an inductively coupled high frequency plasma-optical emission spectrometer of the axial type according to the invention.

This spectrometer of the axial type is essentially similar to the spectrometer described in connection with figure 1 except that the plasma generated by the plasma device 1 is axially oriented with regard to the optical axis OA of the spectrometer detector device 2 and the video-camera 7 is fixed on the enclosure of the plasma generating device 1 in a perpendicular direction to the optical axis of the spectrometer detector device 2. Of course, the video-camera 7 is placed in the enclosure so that the plasma is positioned at a focal point of the video-camera 7.

Obviously, with such a radial plasma spectrometer, the video camera is not able to image the spectrometer detector device entrance slit 3. Otherwise, this embodiment would function and perform as the embodiment of figure 1.

The axial position of the plasma in the embodiment of figure 2 allows replacing the optical emission spectrometer by a mass spectrometer. In that case, relay lens 6 as well as the entrance slit 3 would be replaced by the diaphragm of the mass spectrometer. Otherwise, functioning and results
5 would be similar.

CLAIMS

1. A method for monitoring and/or controlling the status of a plasma in a plasma spectrometer, which comprises :
 - 5 - acquiring image data of the plasma ; and
 - a) displaying an image of the plasma from the image data on a display device, and/or
 - b) storing the image data in a computer unit along with measured data.
- 10 2. The method of claim 1 wherein the image data are processed to obtain intensity images, intensity contour, color contour, intensity outline, time base fluctuation of above image data and compared with a plasma optimal condition data set.
3. The method of claim 1 or 2, wherein the plasma spectrometer is an optical emission spectrometer.
- 15 4. The method of claim 3, wherein the spectrometer has an entrance slit and image data of the spectrometer entrance slit are acquired, and an image of the entrance slit is displayed from the acquired entrance slit image data on the display device simultaneously with the plasma image and/or the acquired entrance slit image data are stored in the computer unit
- 20 simultaneously with the plasma image data
5. The method according to anyone of claims 1 to 4, wherein the plasma is a radially oriented plasma.
6. The method according to anyone of claims 1 to 3, wherein the plasma is an axially oriented plasma.
- 25 7. The method according to anyone of claims 1 to 6, wherein the spectrometer is an optical emission spectrometer.
8. The method according to anyone of claims 1 to 3 and 6, wherein the spectrometer is a mass spectrometer.
9. The method according to anyone of the preceding claims, which
- 30 further comprises acquiring plasma sound data and either reproducing plasma sound from the registered plasma sound data on the display device and/or storing plasma sound data in the computer unit.
10. The method according to anyone of the preceding claims, wherein the display device and/or the computer unit are remotely located
- 35 from the spectrometer.

11. A plasma spectrometer comprising a video-camera (7) fixed on an enclosure of a plasma generating device (1), said video-camera (7) being coupled to a display device (10) and/or computer unit (9) for displaying and/or storing plasma image data obtained through the video-camera.

5 12. A spectrometer of claim 11, wherein the plasma is a radially oriented plasma.

13. The spectrometer of claim 12 wherein image data of an entrance slit (3) of a detector device (2) of the spectrometer are simultaneously obtained with the plasma image data through the video-camera (7).

10 14. The spectrometer of claim 12 or 13, wherein the video-camera (7) is axially positioned with regard to the optical axis of the spectrometer.

15. The spectrometer of claim 11, wherein the plasma is an axially oriented plasma.

16. The spectrometer of claim 15, wherein the video camera is perpendicularly oriented with regard to the optical axis of the spectrometer.

17. The spectrometer according to anyone of claims 11 to 15, which is an optical emission spectrometer.

18. The spectrometer of claim 16, which is a mass spectrometer.

20 19. The spectrometer according to anyone of claims 11 to 18, further comprising a microphone fixed on the enclosure of the plasma generating device (1) for acquiring plasma sound data, said microphone being coupled to the computer unit (9) for storing and/or processing of the plasma sound data.

ABSTRACT

METHOD FOR MONITORING AND / OR CONTROLLING THE
STATUS OF A PLASMA IN A PLASMA SPECTROMETER AND
SPECTROMETER FOR IMPLEMENTING SUCH A METHOD

APPLICANT

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INVENTION

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A method for monitoring and/or controlling the positioning and/or condition of a plasma in a plasma spectrometer, which comprises :

- acquiring image data of the plasma through a video-camera (7), and
- a) displaying on a display device (10) a plasma image from the acquired image data ; and/or
- b) storing the image data in a computer unit (9).

Application to inductively coupled high frequency plasma optical emission and mass spectrometers.

Figure 1

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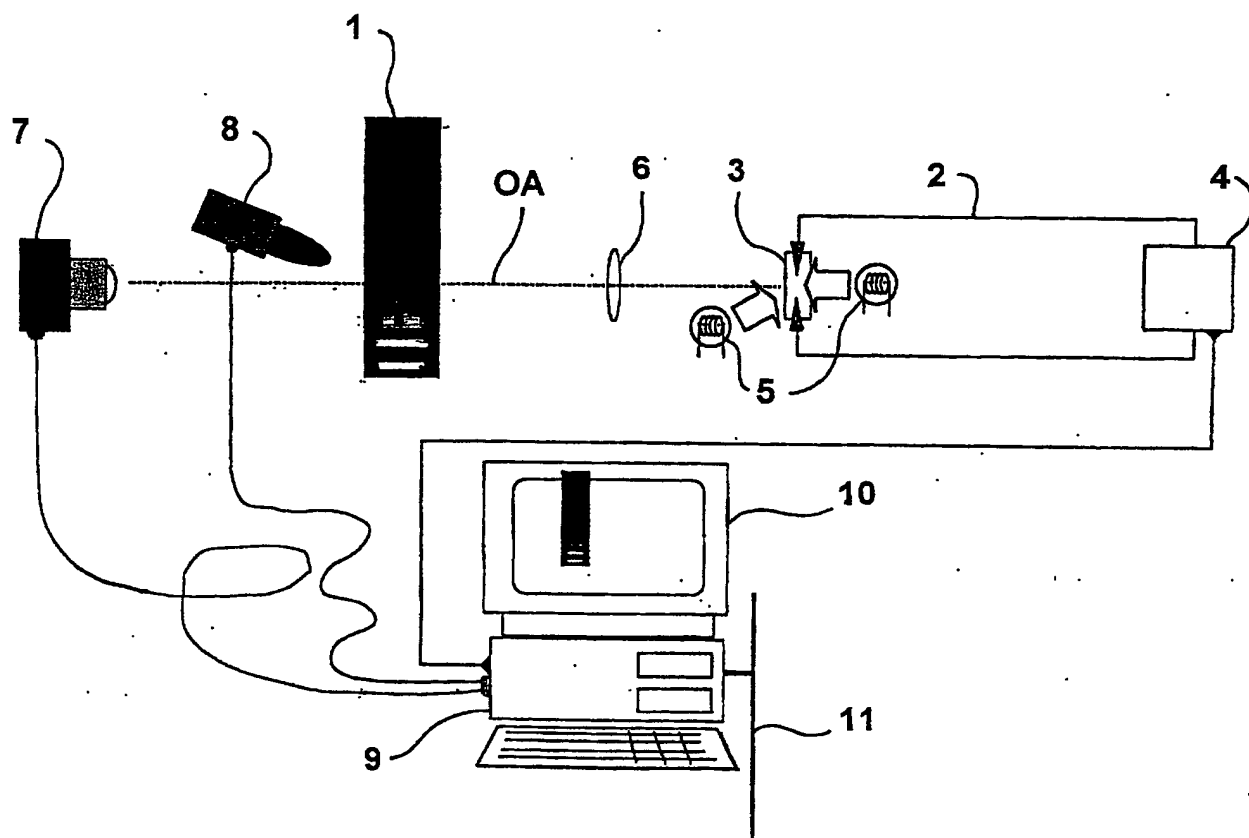


FIGURE 1

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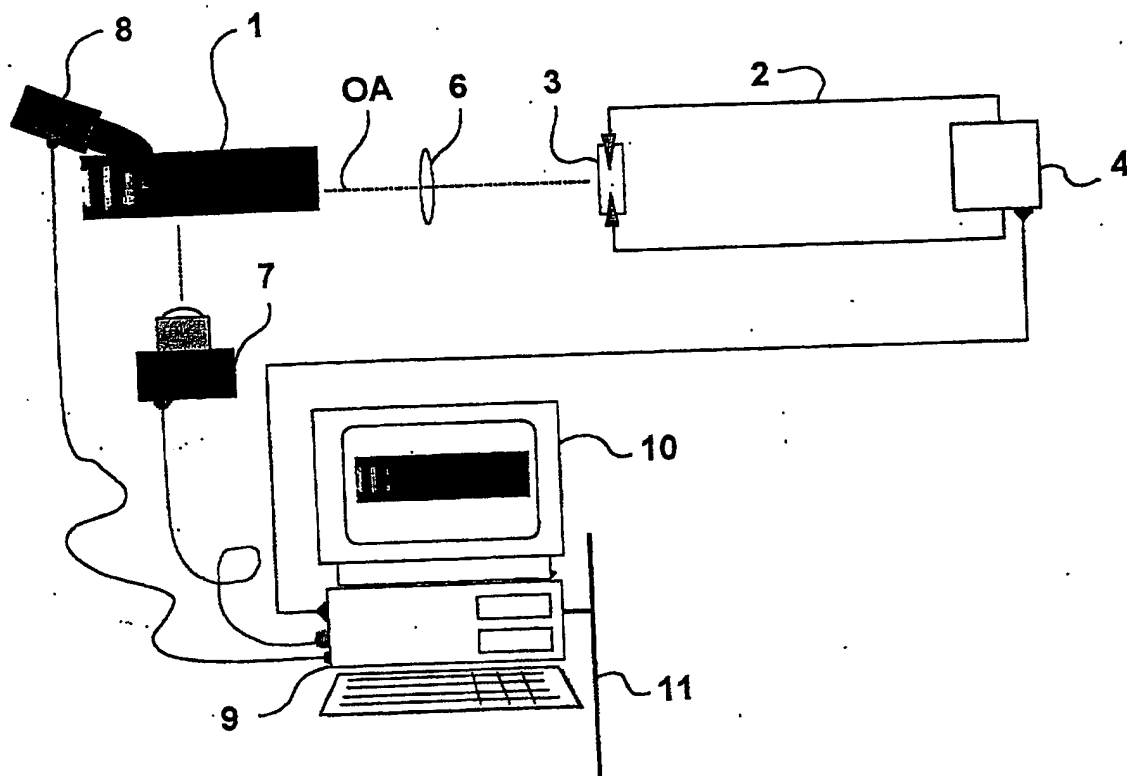


FIGURE 2